Physicochemical treatment (coagulation-flocculation-Fenton) of mature leachates from Tuxtla Gutierrez, Chiapas landfill

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ABSTRACT

Mature leachates are a significant cause of soil and water contamination because they contain high organic loads of recalcitrant materials. The application of a physicochemical process is normally insufficient to remove the organic load from these liquids. The objective of the study was to evaluate removal efficiencies in terms of Chemical Oxygen Demand in mature leachates from the Tuxtla Gutierrez landfill applying a physicochemical Coagulation-Flocculation-Fenton system. In the first stage, ferric chloride ($FeCl_3$) and ferrous sulfate ($FeSO_4$) coagulants were used at different doses (2.2, 2.6 and 2.8 g L^{-1}) and pH values (4, 5 and 6) in a 2 x 3 x 3 experimental arrangement. This effluent was treated in a second stage (Fenton), testing several mass ratios, ranging from 1 to 3, of the oxidant to the catalyst (H_2O_2/Fe^{2+}), maintaining the constant catalyst dose (0.434 g L^{-1} Fe^{2+}). The tests were performed under a 3^2 factorial design at various pH values (2.5, 3.0 and 3.5) and H_2O_2 doses (300, 550 and 800 mg L^{-1}). The oxidation and flocculation stages were performed at 135 rpm for 80 min and at 20 rpm for 20 min, respectively. The highest COD removal (66%) in the coagulation-flocculation process was obtained with 2.2 g L^{-1} of $FeCl_3$ at pH = 6. With the best treatment Fenton reached 70% COD removal at pH 2.5 and 800 mg L^{-1} of H_2O_2. The tests under this physicochemical system are the first application to recalcitrant leachates in Mexico, reaching a 90% overall efficiency and improving the biodegradability index by 64% from 0.14 to 0.23.

INTRODUCTION

The State of Chiapas, Mexico has more than 118 sites for solid waste disposal (commonly known as "rubbish dumps"). More than 95% of them are operated as open-air tips without any control whatsoever, thereby causing harm to the surrounding areas. These sites, whether controlled or uncontrolled, generate gaseous and liquid emissions. Gaseous emissions comprise mainly methane and carbon dioxide, while the liquid emissions come from the decomposition of heterogeneous wastes (leachates). Leachates contain all types of contaminants, many of them in high concentrations, and are thus extremely complex and difficult to treat [1]. Leachates contain high concentrations of organic and inorganic pollutants, including humic acids, ammonium and heavy metals, as well as inorganic salts [2,3].

According to Deng and Englehardt [4], when these liquids are not controlled, they can permeate through the soil reaching underground water supplies or mixing with bodies of surface water, contributing to the contamination of soil and underground and surface water. At the current time, zero leachate management is the

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order of the day in the State of Chiapas, revealing the alarming lack of interest and scant awareness that has prevailed to date regarding one of the most (if not the most) complex and highly contaminating residues continuously generated within the state [5].

Generally speaking, leachate biodegradability is determined based on the biochemical oxygen demand to chemical oxygen demand (BOD/COD) ratio. Wang et al. [6] classify a leachate as poorly biodegradable when the ratio or biodegradability index (BI) ranges from 0 to 0.17; while others consider values below 0.3 [1,7]. This type of leachate is known as old or stabilized leachate. By contrast, when the BI is higher than 0.3, the leachate is considered biodegradable and is known as a young leachate (typically less than two years old) [8]. For this reason, the recommended first treatment stage for young leachates is the application of biological treatments; whereas physicochemical processes are the best option for old leachates [9].

Among the physicochemical treatments the coagulation-flocculation, adsorption with activated carbon, chemical precipitation, processes with membrane and chemical oxidation [10] are preferred with the Advanced Oxidation Processes (AOPs) being especially preferred.

In treating leachates, coagulation has been used as a pre-treatment before the application of biological processes or as a cleaning stage to remove non-biodegradable organic components, or in schemes linked to AOPs [11]. Renou et al. [12] report 15 studies of the coagulation-flocculation process from around the world, in which different coagulants such as aluminum sulfate ($Al_2(SO_4)_3$), ferric chloride ($FeCl_3$) and calcium hydroxide ($Ca(OH)_2$) have been tested, seeking optimum experimental conditions in terms of pH, coagulant dose and mixing speed. The COD removal efficiencies (8-90%) using one or two of these coagulants as well as the operating conditions are varied considerably. In the same way, in the coagulation-flocculation process the removal of other parameters has been evaluated, as presented by Aziz et al. [13] who report high removal efficiencies of suspended solids (95%) and color (90%) with average COD removal efficiencies (43%), by using three types of coagulants.

AOPs have started to be tested in the last two decades with good efficiencies when the organic matter in the leachate is little biodegradable [2,6,13]. For instance, Zhang et al. [14] reports COD removal efficiencies above 60% through the Fenton process while Deng [7] demonstrated COD removal efficiency (> 60%) using a Fenton oxidation process. With regard to the physicochemical systems applied to mature leachates, the AOPs and other physicochemical processes have shown very high efficiency, such as 80% COD removal with an integrated process, Fenton reaction-ultrafiltration [15] and 78% removal in a coagulationozonation integrated system [11].

Based on the above described studies and taking into account the need to treat leachates and their specificity, the objective of this study is to evaluate the efficiency of a physicochemical system (coagulation-flocculation-Fenton), seeking the optimum operating conditions for COD removal and BI evolution, for stabilized leachates of Tuxtla Gutierrez landfill under various operation conditions, and with two different coagulants for the first stage, while the Fenton reaction was used in the second stage. The tests under this physicochemical system represent the first application to recalcitrant leachates in Mexico.

MATERIALS AND METHODS

Monthly, from May to December 2008, leachates were collected from three sumps located in the sealed off area (12 ha) of the landfill in Tuxtla Gutierrez (Fig. 1), located at the geographic coordinates 93°11'59.74" W longitude and 16°39'44.56" N latitude. The samples were refrigerated at 4 °C before tests. The following parameters were determined: BOD, COD, alkalinity (titrated with 0.02 N H_2SO_4) and heavy metals (using an atomic absorption spectrophotometer, VARIAN Spectr-AA 220 under the flame technique). These analyses were performed following the Standard Methods [16].

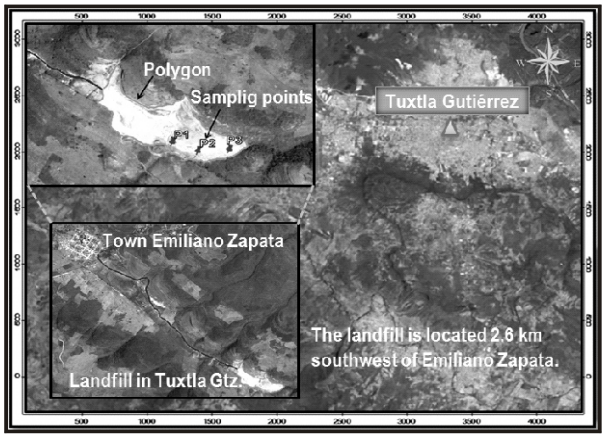

Fig. 1. Location of the sampling points in the landfill of Tuxtla Gutierrez.

The coagulation, flocculation and Fenton System experiments were conducted using JAR TESTER equipment (PHIPPS and BIRD PB700), consisting of 6 jars of 150 mL each whose contents were stirred with flat stirring paddles (18.5 mm x 43.5 mm). Before starting the tests, the refrigerated samples were brought back to room temperature, stirred and filtered to remove large solid particles. Sample volume per jar was 100 mL.

Fermont analytical grade $FeCl_3$ and $FeSO_4$ were used as coagulants. The concentrations of both coagulants (2.2, 2.6 and 2.8 g L^{-1}) were established after performing preliminary tests based on the conditions found by Monje-Ramirez and de Velasquez [11]. The reaction pH values were 4, 5 and 6. Thus, the tests

conducted followed a 2 x 3 x 3 factorial design, giving a total of 18 different treatments. Of each treatment, ten repetitions were made, totaling 180 runs.

Rapid mixing conditions were 80 s at 250 rpm, values close to those used by Méndez Novelo et al. [17]. Slow mixing conditions were 20 min at 30 rpm in order to favor flocs aggregation [18]. Then the samples were allowed to settle for 30 min. After the settling period, volumes of about 10 mL were taken 2 cm below the surface level. The COD was then measured. Initial COD readings in the leachate samples showed a concentration of 2400 ± 100 mg L^{-1}. All the repetitions were adjusted to these values. The COD removal efficiency was calculated based on the initial and final COD concentrations.

The pretreated effluent derived from the most efficient treatment was subjected to a Fenton reaction in a second stage, as described hereinafter.

Preliminary tests identified that the best removal levels were reached with pH values ranging from 2.5 to 3.5. Taking this into account, all the following tests were performed at pH 2.5, 3.0 and 3.5. 10% sulfuric acid (H_2SO_4) was used for pH adjustment purposes. With regard to the Fenton reagents, various oxidant to catalyst (H_2O_2/Fe^{2+}) mass ratios were used, ranging from 1 to 3, at a constant catalyst dose (0.434 g L^{-1} Fe^{2+}). In order to establish the most appropriate conditions for COD removal and taking into account the oxidant and the pH as factors, three level experiments were run in a 3^2 factorial design. Ten repetitions were made of each treatment, totaling 90 runs.

In the oxidation stage, the jar test equipment was adjusted at a stirring speed of 135 rpm for 80 min. Then, the pH of the solution was adjusted to 7 to 8 with 2 M NaOH to facilitate the flocculation process that was performed for 20 min at 20 rpm.

The flocs formed were separated for 1 h in a test tube. Afterwards, about 10 mL of sample were taken 2 cm below the surface level, and warmed in a water bath for 30 min at 50 °C to remove any residual H_2O_2 from the solution [7]. Once this sample reached room temperature, COD and TOC were determined. Finally, the results generated in both stages were analyzed through a 2-way variance using the JMP 5.1 statistics package.

RESULTS AND DISCUSSION

In 2006, the site in question was still operating and Cisneros et al. [19] performed monthly samplings of the leachates produced there. For the current project, a series of samples were taken, throughout an 8-month period in 2008, when the site had already been closed. The results of both periods are shown in Table 1.

For both periods, the results show BI below 0.3, important index because it defines to a great extent the leachate type and treatment to which it has to be submitted. According to others [1,6,7], values below 0.3 refer to a mature or type III leachate and thus it can be classified as little biodegradable [13] with the presence of partially stable and biologically resistant recalcitrant organic materials, such as a humic and fulvic acid as well as various salts [2]. Wang et al. [6] essentially report the similar characteristics of mature leachates in more than 20 works performed in different parts of the world, with BI below 0.3 and pH and alkalinity values ranging from 6.8 to 9.0 and 1020 to 12300 mg L^{-1} of $CaCO_3$, respectively.

With regard to the presence of Total Suspended Solids (TSS), in this study a relatively high average concentration (400-600 mg L^{-1}) was found, compared to the values (280 to 320 mg L^{-1}) observed for mature leachates by others [13,15,20], but was comparable to the 550 mg L^{-1} for the leachates from Shanghai landfill [21]. It is very difficult to establish a range for the TSS because its concentration depends on various factors, such as residue composition, compaction level and cover material characteristics, among others.

The heavy metal levels showed some similarity with those reported for other landfill in methanogenic

Table 1. Characteristics of the leachates from the sealed off area of the landfill in Tuxtla Gutierrez, Chiapas, Mexico

Parameters	2006 [19]				2008			
	Concentration (mg L^{-1})			Standard Deviation	Concentration (mg L^{-1})			Standard Deviation
	Average	Minimum	Maximum		Average	Minimum	Maximum	
pH		7.7	8.42			8.4	8.8	
BI	0.16	0.01	0.47	0.18	0.15	0.02	0.19	0.05
Alkalinity	10010	6780	14610	2010	9130	8550	9770	428
TSS	5593	75	1223	385	503	396	610	152
Iron	31.6	17.8	56.5	14.0	21.2	17.1	25.2	5.7
Nickel	1.09	0.79	1.49	0.19	1.64	1.4	1.88	0.34
Lead	0.24	0.17	0.36	0.06	1.85	0.48	3.23	1.94
Zinc	1.63	0.81	2.35	0.53	3.43	2.45	4.41	1.39
Copper	0.12	0.09	0.14	0.02	0.19	0.14	0.24	0.06
Cadmium	0.018	0.014	0.028	0.004	nr	nr	nr	nr
Chromium	1.92	1.07	2.87	0.67	2.73	1.96	3.49	1.08

phase (Table 2). However, the high iron values registered for the present study (17-25 mg L^{-1}) were above the values (6.5-13.2 mg L^{-1}) found in other landfills around the world [2,22,23,25]. According to Nájera et al. [5], the rocks that abound in the sealed off area of the landfill in Tuxtla Gutierrez result from interactions between sandstone and limonites, and between shale and clayey limestone. As limonites are a group of iron minerals, this could explain the higher concentrations of iron in the leachate. Thus, high iron levels in the Tuxtla Gutierrez landfill leachate should not be considered in any way as an industrial source.

Table 2. Heavy metal concentration in mature leachates from different landfills

Parameter (mg L^{-1})	Landfill (average values)				
	Curitiba (Brazil) [2]	Mer, Yuc. (Mexico) [22]	Landfill sites (Swedish) [23]	Jebel Chakir (Túnez) [24]	Shalu (Taichung, Taiwan) [25]
Iron	13.2	8.5	6.5	nr	8.1
Nickel	1.43	0.35	0.03	0.53	0.17
Lead	0.28	0.03	< MDL	nr	< MDL
Zinc	1.06	0.59	0.07	0.94	0.31
Copper	0.36	0.06	0.02	0.14	0.23
Cadmium	nr	< MDL	< MDL	nr	< MDL
Chromium	0.45	nr	0.02	2.24	0.77

nr = not reported; MDL = method detection limit (0.01 mg L^{-1})

Based on the above and specifically with regard to the basic parameters (pH, alkalinity, BI, etc.) [6], it was defined as a type III leachate, better known as mature or old leachate. Others [4,9] have recommend physicochemical processes as first stage for the treatment of said liquids and thus, the leachate in question was treated with a physicochemical system, coagulation-flocculation using $FeSO_4$ and $FeCl_3$ in the first stage, and Fenton reaction in the second stage.

For the first stage, the average removal efficiencies obtained in each treatment and per coagulant are shown in Table 3 based on a 2 x 3 x 3 experimental arrangement. The results of Table 3 show that all the $FeCl_3$ treatments reached removal levels above 40%, much higher than the ones registered with $FeSO_4$ (around 10%). Among the tested pH values, pH 6 exhibited the highest removal efficiencies (66%) at the lowest $FeCl_3$ dose (2.2 g L^{-1}). These results coincide partially with the ones reported by Aziz et al. [13] with regard to the superiority of $FeCl_3$ versus $FeSO_4$ at pH 6. However, the best COD removal efficiency reported by Aziz et al. [13] was 45% at initial COD level of 2980 mg L^{-1} (coagulant 2.5 g L^{-1}). Nonetheless, our results are similar to those by Monje-Ramirez and de Velasquez [11] who reported a 67% COD removal efficiency using $FeCl_3$ as coagulant, evidencing the effectiveness of the coagulation-flocculation process in the removal of the organic load from mature leachates as well as the viability of its application as pre-treatment stage within a physicochemical system, as various researchers have shown [12,26-28].

Table 3. COD Removal efficiencies (%) for each treatment in the coagulation-flocculation process

	$FeCl_3$			$FeSO_4$		
Dose (g L^{-1})	pH 4	pH 5	pH 6	pH 4	pH 5	pH 6
2.2	43 ± 3	48 ± 3	66 ± 3	8 ± 1	10 ± 1	9 ± 1
2.6	46 ± 4	48 ± 5	65 ± 3	12 ± 2	10 ± 1	14 ± 1
2.8	45 ± 5	51 ± 3	60 ± 5	10 ± 1	14 ± 2	12 ± 1

The major effectiveness of $FeCl_3$ in the coagulation-flocculation process can be attributed to the fact that colloidal particles precipitate better with higher charge electrolytes [29]. For this reason the hydroxide-metal complexes formed by Fe^{3+} according to Eq. 1 [30] can be more efficient than the hydroxide-metal complexes formed by Fe^{2+}, and thus the insoluble hydroxide formed trap the neutralized colloids facilitating their removal. The acid formed reacts with the bicarbonate alkalinity in the leachate. The above explains why the flocs formed upon using $FeCl_3$ are larger than the ones obtained using $FeSO_4$, as shown by the optic microscopy studies performed by Soto Regalado et al. [31].

$$FeCl_3 + 3H_2O \rightarrow Fe(OH)_3 + 3H^+ + 3Cl^- \quad (1)$$

Moreover, a variance analysis was conducted (for major coagulant) and it was observed that 60% (n = 90) of the data showed variability and that pH 6 was significantly different ($p = 3.3 \times 10^{-19}$) from the other two levels (Fig. 2a), that also occurred with regard to the interaction between treatments (Fig. 2b), upon observing that all combinations at pH 6 showed significant differences (p = 0.137) versus the rest of the combinations at pH 4 and 5. However, in the case of the doses used, there were no significant differences (p = 0.741) among the three dose levels. Thus, the best removal efficiencies were obtained at pH 6 with the lowest coagulant concentration (2.2 g L^{-1}).

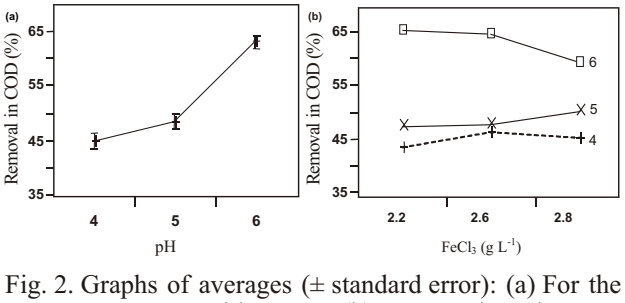

Fig. 2. Graphs of averages (± standard error): (a) For the treatment with pH; (b) Interaction between treatments.

With regard to the BI determined on the effluent of the best treatment, no changes were observed and it remained at 0.14 as at the beginning of the process. This can be attributed to the fact that in the coagulation process the colloidal particles that are mostly removed are humic substances [32] and it can thus be expected that there will not be recalcitrant compound transformation to more biodegradable forms.

The effluent of best treatment with $FeCl_3$ (2.2 g L^{-1}) was subjected to a second stage Fenton reaction. The average results obtained for each treatment are shown in Table 4 with a 3^2 factorial design. The results show that the best removal efficiencies were obtained with the lowest pH value (2.5) for all the treatments, obtaining the best COD removal efficiency (70%) at the highest oxidant dose (800 mg L^{-1}). As in the case of the first stage, the pH showed a strong influence on the process.

Table 4. Effect of pH and H_2O_2 dose on removal efficiency in terms of COD in the Fenton process

pH	H_2O_2 Dose (mg L^{-1})		
	300	550	800
	Removal efficiency in term of COD (%)		
2.5	49 ± 8	49 ± 9	70 ± 3
3.0	25 ± 6	30 ± 7	40 ± 8
3.5	25 ± 8	26 ± 4	34 ± 7

The low pH value is outside the range (3.0 to 6.0) reported by Wang et al. [6] and outside the optimum values (pH = 3) reported by others [33,34] but coincides with the value reported by Zhang et al. [14]. Moreover, Zhang et al. [14] also reported the pH is one of the most influential factors in the Fenton process. Thus, if the pH is below its optimum value, the process oxidation can be inhibited because of the H^+ excess competition for hydroxyl radicals (•OH) [35].

If the pH is above the optimum value, especially for values close to neutral, even more inhibition mechanisms can occur. One of the main inhibition mechanisms is related to the competing effect exhibited by some compounds such as HCO_3^- because it reacts easily with •OH according to Eq. 2 [36].

$$\bullet OH + HCO_3^{=} \rightarrow H_2O + \bullet CO_3^{2-} \quad (2)$$

With regard to the H_2O_2/Fe^{2+} ratio, it can be observed that the best results are reached when the molar ratio was 3, which is in agreement with that reported by Deng [7] who comments that the oxidation efficiency of the Fenton process diminishes when the molar ratio is < 3 owing to the increase of the competing effect of Fe^{2+} for •OH, as shown in Eq. 3.

$$\bullet OH + Fe^{2+} \rightarrow Fe^{3+} + OH^- \quad (3)$$

Overall, the removal efficiency (70%) was relatively high compared to those reported in the literature for mature leachates under the Fenton process, e.g., 61% COD removal [7,14] or 69% [37].

In this stage, a variance analysis was performed, in which it was found that 78% (n = 90) of the data presented variability, and that the H_2O_2 level at 800 mg L^{-1} ($p = 1.55 \times 10^{-26}$) at pH 2.5 was significantly different ($p = 2.43 \times 10^{-11}$) from other levels. In the case of the interaction between treatments, Fig. 3 shows that all the combinations at pH 2.5 showed significant differences (p = 0.172) compared to the other combinations at pH 3 and 3.5. The best removal efficiencies were obtained at pH 2.5 and with the highest oxidant dose.

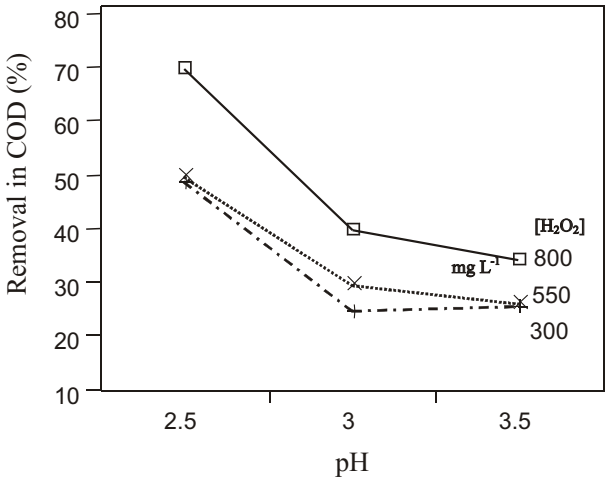

Fig. 3. Graphs of averages (± standard error) for the interaction between treatments.

With regard to BI, it is known that the Fenton reaction promotes the generation of hydroxyl radicals (•OH) [6], a highly oxidant species having the capacity to attack virtually any recalcitrant molecule [2] and transform it into less complex and more biodegradable molecules [11], increasing thus leachate biodegradability [4]. This is indeed the case in our study as BI increases from 0.14 to 0.23, i.e., a 64% improvement. Upon comparing this result with the other two studies (Table 5) it can be seen that it is similar to the values obtained by Kim et al. [34] and higher than the values reported by Méndez Novelo et al. [17] and Méndez Novelo et al. [38]. However, the index increase was relatively low compared to the values reported by Lopez et al. [39] whose final BI increased by 150%, or from 0.2 to 0.5.

Table 5. Biodegradability index in mature leachate after the application of advanced oxidation processes

AOP	BI_o	BI_f	Reference
H_2O_2/Fe^{2+}	--	0.22	[34]
H_2O_2/Fe^{2+}	0.07	0.10	[17]
H_2O_2/Fe^{2+}	0.07	0.13	[38]
H_2O_2/Fe^{2+}	0.20	0.50	[29]

AOP: Advanced Oxidation Process; BI_0 and BI_f: initial and final Biodegradability Index, respectively.

The overall COD removal efficiency under the coagulation-flocculation-Fenton coupled system tested was 90%, slightly higher than the values reported for other coupled systems, such as 73% removal in coagulation-Fenton system [40], 78% efficiency in coagulation-ozonation system [11], and 80% removal under an integrated scheme of Fenton-ultrafiltration [15].

CONCLUSIONS

The process of coagulation-flocculation applied to mature leachates of the landfill in Tuxtla Gutierrez demonstrated that the process was effective in COD removal using $FeCl_3$; and that the best removal efficiencies (66%) were obtained at pH 6 using a dose of 2.2 g L^{-1}, at 250 and 30 rpm for fast and slow mixing, respectively. When $FeSO_4$ was used as the coagulant, the COD removal was poor, with only 10% removal on average.

The Fenton process proved to be efficient in COD removal (70%) with efficiencies obtained being comparable to the highest values reported in the literature. Thus, this work shows that the coagulation-flocculation-Fenton physicochemical system can be applied to the treatment of mature leachate of the area of study with 90% COD removal efficiency. This finding is relevant given that it is the first experience of leachate treatability in the state of Chiapas, and the first test applied to recalcitrant leachates in Mexico under this physicochemical system.

Based on the BI value, a constant value is observed in the coagulation-flocculation process, since the removal of the organic load can be attributed basically to humic colloidal particles and chemical structures of which are not subjected to transformation. The Fenton process makes it possible to transform the recalcitrant materials to more biodegradable forms permitting the improvement of the leachate BI from 0.14 to 0.23, suitable to a final biological system.

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